



CASE PR/3-23156/A/RAI 56/PCT

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF
KENNETH SUNDBERG ET AL.
INTERNATIONAL APPLICATION NO. PCT/EP 03/02534
FILED: JUNE 12, 2003
FOR: PAPER SIZING COMPOSITION
U.S. APPLICATION NO: 10/517,413
35 USC 371 DATE: December 3, 2004

Group Art Unit: 1731
Examiner: Dennis R. Cordray

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 1.132

I, Dr. Kenneth Sundberg, a citizen of Finland and a resident of Turku, Finland hereby declare:
That I was awarded a Doctor of Science in 1995.

That I was an employed of Ciba Specialty Chemicals Oy (formerly Raisio Chemicals Oy) as a
Research Manager in the field of paper Sizing agents and their applications since 1998.

I am one of the inventors listed on the above application and have overseen the experimental work
reflected in the tables 1, 2 and 3 disclosed below.

Objective:

To see the effect of olefin and polymeric residue content on the ASA sizing performance.

Distillation:

Alkenyl succinic anhydride (C16-18 ASA) was distilled. Three fractions were collected and the samples were analyzed.

Table 1. The analysis of the four ASA samples.

No	Sample	Olefin (%)	ASA (%)	Polymeric residue (%)
1	ASA	1.7	84.5	13.4
2	First fraction	10.3	88.9	-
3	Second fraction	-	100.0	-
4	Third fraction	-	27.8	72.2

Experiment:

1. ASA samples with different olefin contents (0 - 7 %) and polymeric residue contents (0 – 25 %) were prepared as sizing agents in the experiment. A conventional ASA (No 1) and a distilled ASA (No 3) were used as reference.
2. The sizing evaluation was carried out using a solvent sizing procedure. A standard furnish (30 % softwood and 70 % hardwood) was used to prepare unsized paper samples. Acetone was used as solvent.
3. Solutions of the designed ASA's were prepared at different concentrations. Then unsized papers were immersed into these solutions for 1 hour. Excess solvent was removed and the papers were then dried in an oven at 105 °C for 1 hour. Cobb tests were performed on each sized paper.

Results and discussions:**1. The effect of the concentration**

Table 1. The sizing comparison at concentration of 650 mg/L

No	Sample	Content (%)			Cobb (g/m ²)
		Olefin	ASA	Polymeric residue	
0					128.5
1	Conventional ASA	1.7	84.5	13.4	50.6
2	First fraction	10.3	88.9	-	85.7
3	Distilled ASA	-	100.0	-	36.2

4	Final fraction	-	27.8	72.2	53.1
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At a soft sizing, distilled ASA had a better sizing efficiency than the parent conventional ASA (No 1) and both ASA with high impurity (No 2 with olefin and No 4 with polymeric residue).

Table 2. The sizing comparison at concentration of 1000 mg/L

No	Sample	Content (%)			Cobb (g/m ²)
		Olefin	ASA	Polymeric residue	
0					128.5
1	Conventional ASA	1.7	84.5	13.4	45.2
2	First fraction	10.3	88.9	-	88.7
3	Distilled ASA	-	100.0	-	25.1
4	Final fraction	-	27.8	72.2	107.0

At a standard sizing (reference Cobb = 25 g/m²), distilled ASA had a better sizing efficiency.

Table 3. The sizing comparison at concentration of 1400 mg/L

No	Sample	Content (%)			Cobb (g/m ²)
		Olefin	ASA	Polymeric residue	
0					128.5
1	Conventional ASA	1.7	84.5	13.4	20.7
2	First fraction	10.3	88.9	-	23.6
3	Distilled ASA	-	100.0	-	20.2
4	Final fraction	-	27.8	72.2	24.8

At a hard sizing, distilled ASA, again had a better sizing efficiency. However, the difference is not so obvious as at a standard sizing.

Therefore, in the late experiment, the size solution of 1000 mg/L was used.

2. The effect of olefin content

The results in table 4 below are from various blends of #2 and #3 in Table 1 to give the % olefin content in ASA below.

Table 4. The effect of olefin content (at 1000 mg/L)

No	Olefin content (%)	Cobb (g/m ²)
3	0	25.1
10	1	30.2
11	2	40.2
12	3	56.4
13	5	64.2
14	7	83.5
2	10.3	88.7

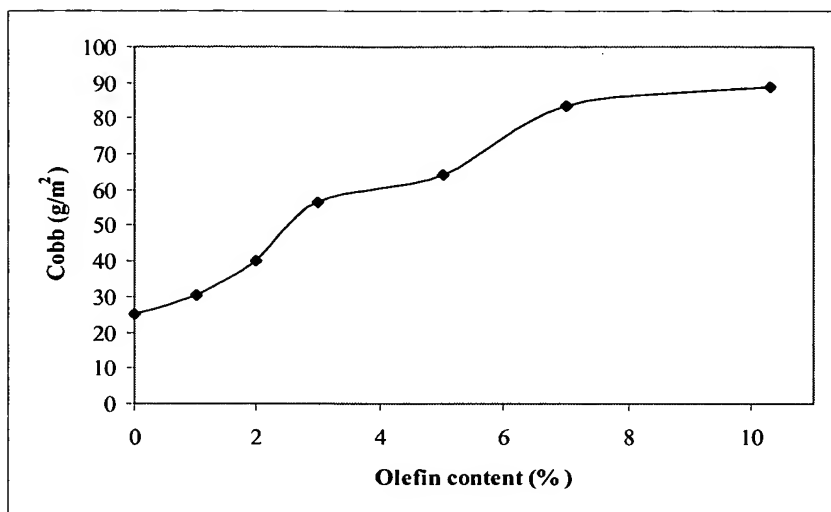


Figure 1. The effect of olefin content on the sizing

It is clear from table 4 and figure 1 that the sizing efficiency decreased when the olefin content increased.

3. The effect of the polymeric residue

The results in table 4 below are from various blends of #3 and #4 in Table 1 to give the % polymeric residue content in ASA below.

Table 4. The effect of polymeric residue (at 1000 mg/L)

No	Polymeric residue content (%)	Cobb (g/m ²)
3	0	25.1
5	5	39.4
6	10	50.2
7	15	49.4
8	20	46.4
9	25	53.0
4	72.2	107.0

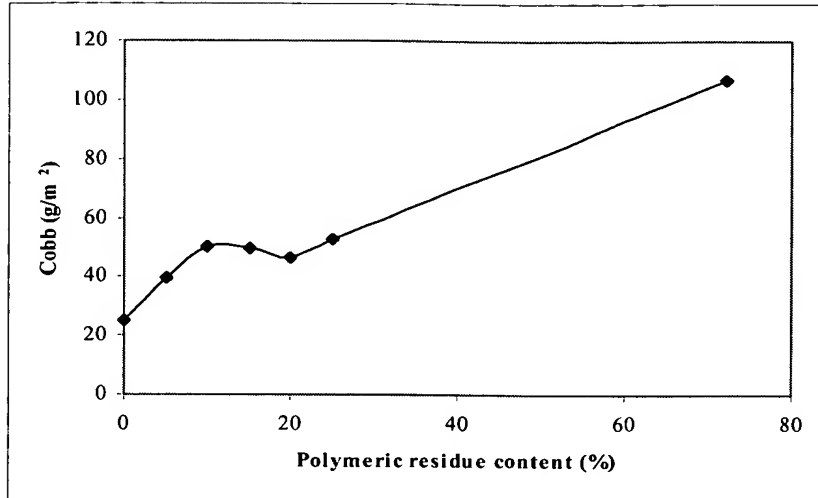


Figure 2. The effect of the polymeric residue on the sizing.

It is clear from table 5 and figure 2 that the sizing efficiency decreased when the polymeric residue content increased.

Conclusion:

- ASA sizing efficiency decreased when the olefin content and polymeric residue content increased.
- Furthermore, the examiner has commented on page 4, second paragraph of 01/22/2008 Office action "that the Specification does not indicate how the ASA was applied to the papers, whether by front end addition, size press, or by some other method of application". This is incorrect. The applicants clearly say that ASA emulsified with starch was dosed via a tube in the accepting side of the mixing box and the other tube nearby is used to add cationic wet end starch. From the mixing box the pulp goes to the head box which doses it to the forming section. See page 10, third paragraph. Thus the applicants clearly disclose wet end addition of the ASA in the specification example.
- The examiner believes the applicants to have failed to provide sufficient data in the specification to support broadly stated claims that embody a nonspecific paper or board of any weight made from any pulp, having any additives and any amount of ASA applied in the wet end, via size press or by any other method of application.

- As one skilled in the art of papermaking, I would not expect an ASA sizing of the claimed polymeric residue and olefin content to show a trend (that is Cobb values which do not improve) which differs from that shown in the declaration and specification when:
 - the paper or board is characterized by a different pulp makeup,
 - the paper or board is a different weight,
 - the ASA is applied via size press or
 - when the amount of ASA is varied.
- There are several points expressed by the examiner which as a person skilled in the art of papermaking, I believe to be incorrect. The examiner believes it to be desirable to use purified products. However, in papermaking one skilled in the art tries to use inexpensive raw materials unless there is a known advantage in using more highly purified and thus more costly alternatives. Conventional ASA is known to provide sufficient sizing effect. However, the present inventors have discovered that the presence of low amounts of polymeric residues and olefinic content in ASA gives a clear sizing advantage. As this advantage was previously unknown the use of purified ASA would not be considered because of its increased cost.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 26th day of March, 2008



Dr. Kenneth Sundberg